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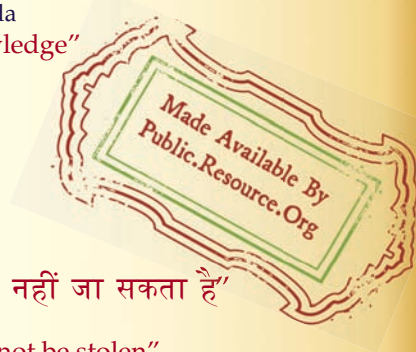
IS 8494 (1977): MCPA, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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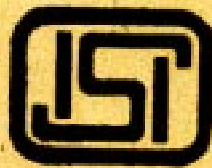
IS : 8494 - 1977

(Reaffirmed 2002)

Indian Standard

SPECIFICATION FOR MCPA, TECHNICAL

UDC 632.954 MCP



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INDIAN STANDARDS INSTITUTION

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NEW DELHI 110002

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September 1977

Indian Standard

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Indian Standard

SPECIFICATION FOR MCPA, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 June 1977, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

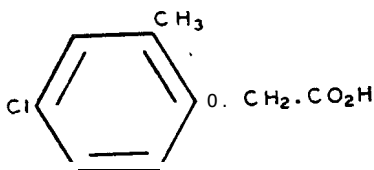
0.2 MCPA is a hormone-type herbicide readily absorbed by leaves and roots and translocated, and used for the control of annual and perennial weeds in cereals, grassland and turf.

0.3 MCPA is the accepted common name by the International Organization for Standardization (ISO) for 4-chloro-2-methylphenoxyacetic acid, also known as 4-chloro-*O*-cresoxyacetic acid and MCP. The empirical and structural formula and the molecular mass of MCPA are as indicated below:

Empirical Formula
 $C_9H_9ClO_3$

Structural Formula

Molecular Mass
200.5



0.4 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS :2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for MCPA, technical.

2. REQUIREMENTS

2.1 Description — The material shall essentially comprise 4-chloro-2-methylphenoxyacetic acid and shall be in the form of white to brown, granular solid, free-flowing powder with a slight odour.

2.2 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR MCPA, TECHNICAL
(Clauses 2.2 and 5.1)

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO	
			Appendix of this Standard	Appendix of IS : 8294-1976*
(1)	(2)	(3)	(4)	(5)
i)	Extractable acids including MCPA (expressed as 4-chloro-2-methylphenoxy-acetic acid), percent by mass, <i>Min</i>	94	—	A
ii)	MCPA, percent by mass, <i>Min</i>	70	A	—
iii)	Free phenols (expressed as 4-chloro-2-methyl-phenol), percent by mass, <i>Max</i>	1.5	—	B
iv)	Material insoluble in NaOH, percent by mass, <i>Max</i>	0.05	B	—
v)	Sulphated ash, percent by mass, <i>Max</i>	1.0	c	—

*Specification for liquid amine salt of MCPA.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry containers made of mild steel. For packs upto 10 kg, tinplate containers may be used. The containers shall also comply with the general requirements as stipulated in 2 of IS : 8190 (Part I)-1976*.

*Requirement for packing of pesticides: Part I Solid pesticides.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the provisions required under the Insecticides Act and Rules:

- a) Name of the material;
- b) Name of the manufacturer;
- c) Batch number;
- d) Date of manufacture;
- e) MCPA content, including the extractable acids percent (m/m);
- f) Net mass of contents; and
- g) The cautionary notice worded as in Insecticides Act and Rules.

3.2.1 Each container may also be marked with the **ISI** Certification Mark.

NOTE — The use of the **ISI** Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The **ISI** Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by **ISI** and operated by the producer. **ISI** marked products are also continuously checked by **ISI** for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the **ISI** Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods of sampling for pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published the matter shall be subject to agreement between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS:1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (*second revision*).

APPENDIX A

[Table 1, Item (ii)]

DETERMINATION OF MCPA CONTENT

A-1. PRINCIPLE

A-1.1 MCPA (4-chloro-2-methylphenoxyacetic acid) is separated from associated acids by partition chromatography on a celite column, and is determined by titration with sodium hydroxide.

A-2. APPARATUS

A-2.1 Chromatographic Column — 65 cm long, internal diameter 10 to 11 mm, fitted at the lower end with a stop-cock and at the upper end with a ground glass socket to take a separating funnel.

A-2.2 Separating Funnel — 500 ml capacity, to fit the column.

A-3. REAGENTS

A-3.1 Phosphate Buffer — pH 6.6. Prepared by dissolving 34 g potassium dihydrogen orthophosphate in about 50 ml water. Add 39 ml of sodium hydroxide solution (1 N) and 300 ml water. Check the pH of the solution, and adjust, if necessary, with more sodium hydroxide. Make up to one litre with water.

A-3.2 Diethyl Ether

A-3.3 Cyclohexane

A-3.4 Tertiary Amyl Alcohol

A-3.5 Eluents

A-3.5.1 Diethyl Ether — Cyclohexane Mixture (A) — 1 + 1 by volume. Equilibrate the mixture (1 litre) by shaking with 25 ml phosphate buffer in a separating funnel. Filter after equilibration to remove suspended droplets.

A-3.5.2 Diet/\$ Ether (B) — Equilibrate as in B-3.5.1. The aqueous phase is used for buffering the support.

A-3.5.3 Diethyl Ether — Amyl Alcohol (C) — 95 : 5 by volume. Equilibrate as in B-3.5.1.

A-3.5.4 Diethyl Ether — Amyl Alcohol (D) — 92 : 8 by volume. Equilibrate as in B-3.5.1.

A-3.6 Bromothymol Blue — 0.04 percent, neutral aqueous solution.

A-3.7 Standard Sodium Hydroxide Solution — 0.005 N.

A-3.8 Silica Gel — of such fineness that it passes through a 125-micron IS Sieve but is retained on 75-micron IS Sieve (see IS : 460-1962*). Heat with 5 N hydrochloric acid on a boiling water-bath for 10 hours, and then wash with distilled water until free from chloride ions. Dry at 140°C for 4 hours.

A-4. PROCEDURE

A-4.1 Preparation of the Column — Mix well, 20 g of the prepared silica gel in small portions with the buffer solution, and make into a paste by small additions of eluent A. Plug the bottom of the column with a small plug of glass wool, half fill the tube with eluent A, and then add the prepared paste in small portions, carrying down the decanted material with the packing disc. After each addition apply gas pressure to the top of the column to assist in the removal of the solvent.

The silica gel shall always remain covered with eluent. In case, level of eluent falls below the level of the silica gel, the column shall be changed. The total length of the finished silica gel column should be between 55 and 60 cm. Cover the top of the silica gel with a circle of filter paper.

A-4.2 'Determination of MCPA

A-4.2.1 Weigh, accurately, sufficient quantity of the sample to contain about 0.7 g of MCPA. Dissolve in eluent A in the volumetric flask, and then make up to 100 ml (solution M).

A-4.2.2 Allow the level of the liquid in the column to fall to about 2 or 3 mm above the silica gel. Add, from the pipette, delivering between two graduations, 1 ml of solution M. Again allow the level to fall to 2 or 3 mm above the silica gel, wash the inner walls of the column with 1 ml of eluent A; and repeat with a further 1 ml portion. Put 125 ml eluent A into the top funnel, and assemble the apparatus. Eluate, until the first acid (4,6-dichloro-2-methylphenoxyacetic acid) has been eluated, collecting the eluate in 4 to 5 ml fractions in the conical flasks, which contain 4 ml water coloured blue by 3 drops of bromothymol blue. The rate of eluation should be about 45 ml per hour. After the first acid has been eluated collect three blank fractions and if any eluent A remains in the separating funnel, detach the funnel, and remove surplus eluent A from the column. When not more than 2 mm of eluent A remains above the silica gel, attach a separating funnel containing 100 ml of eluent B and continue the eluation until all the 4-chloro-2-methylphenoxy-acetic acid has left the column. Remove the funnel of eluant B and any surplus eluent B as before, replace with a separating funnel containing 50 ml of eluent C and eluate until all the third acid (6-chloro-2-methylphenoxyacetic acid) has been eluated. Finish the eluation with 100 ml of eluent D.

*Specification for test sieves (revised).

A-4.2.3 As soon as it has been collected, titrate each fraction with the standard sodium hydroxide solution, to a clear blue end point. During the titration pass a current of air (free from carbon dioxide) or nitrogen through the liquid, and stir the liquid magnetically to ensure thorough mixing of the two phases.

NOTE-The first fraction needs only a drop of standard sodium hydroxide solution. The arrival of the 4, 6-dichloro-2-methylphenoxyacetic acid from the 4-chloro-2-methylphenoxyacetic acid at the bottom of the column is shown by the increased volumes of sodium hydroxide required, which rise to a peak and then die away to a minimum marking the separation of the 4, 6-dichloro-2-methylphenoxyacetic acid from the 4-chloro-2-methylphenoxyacetic acid. There may be two peaks for the 4-chloro-2-methylphenoxyacetic acid, the second lagging behind owing to the late arrival of eluent B. Similarly for 6-chloro-2-methylphenoxyacetic acid and, finally, for the 2-methylphenoxyacetic acid.

A-4.2.4 Construct the chromatogram by plotting the number of the fractions along the abscissa and the volumes of the sodium hydroxide used along the ordinate. Determine the quantity of standard sodium hydroxide solution corresponding to each acid. A base line correction should be made while taking the sum of the titres.

A-4.3 Calculations

$$\text{MCPA, percent by mass} = \frac{2\,006\,VN}{M}$$

where

V = volume in ml of the standard sodium hydroxide solution corresponding to 4-chloro-2-methylphenoxyacetic acid,

N = normality of standard sodium hydroxide solution, and

M = mass in g of the sample taken for the test,

APPENDIX B

[Table 1, Item (iv)]

DETERMINATION OF MATERIAL INSOLUBLE IN SODIUM HYDROXIDE

B-1. REAGENT

B-1.1 Sodium Hydroxide Solution — 0.05 N.

B-2. PROCEDURE

B-2.1 Weigh accurately 1 g of the sample and transfer to a 30-ml stoppered test tube. Dissolve the material using 10 ml sodium hydroxide solution, by shaking. Examine the resulting solution to see whether it is

free from appreciable sediment. Shake the solution and pour it through a 150-micron IS Sieve (see IS : 460-1 962*), washing out any residue from the test tube with water on to the sieve. Wash the residue on the sieve several times with distilled water and allow to drain. Brush the residue remaining on the sieve on to the glazed paper and then to the tared weighing bottle. Dry the weighing bottle and residue at 100°C, cool and reweigh. Calculate the percent by mass of material insoluble in sodium hydroxide.

A P P E N D I X C

[Table 1, Item (v)]

DETERMINATION OF SULPHATED ASH

C-1. PROCEDURE

C-1.1 Weigh accurately about 5 g of the sample into a tared silica crucible with minimum dimensions of 51 X 51 mm. Moisten well with 95 percent ethanol (or industrial methylated spirit) and add 5 drops of concentrated sulphuric acid. Evaporate to dryness over a period of 1 to 2 h by heating *gently* on a gauze or asbestos board, avoiding sputtering. Ignite until most of the carbon is burnt off. Allow to cool, add a few more drops of concentrated sulphuric acid, and re-ignite to constant mass.

C-2. CALCULATION

C-2.1 Sulphated ash, percent by mass = $\frac{100\ m}{M}$

where

m = mass in g of the sulphated ash, and

M = mass in g of the material taken for the test.

*Specification for test sieves (*revised*).

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IS :

560-1969	BHC, technical and refined (second revision)
563-1973	DDT, technical (second <i>revision</i>)
1051-1973	Pyrethrum extracts (<i>first revision</i>)
1251-1973	Zinc phosphide, technical (<i>first revision</i>)
1306-1974	Aldrin, technical (<i>first revision</i>)
1309-1974	Endrin, technical (<i>first revision</i>)
1486-1969	Copper oxychloride, technical (<i>first revision</i>)
1682-1973	Cuprous oxide, technical (fungicidal grade) (<i>first revision</i>)
1832-1961	Malathion, technical
1833-1961	Diazinon, technical
2125-1962	Phenyl mercury salicylate, technical
2126-1973	Phenyl mercury acetate, technical (<i>first revision</i>)
2127-1962	Stabilized methoxy ethyl mercury chloride concentrate
2128-1973	Parathion ethyl, technical (<i>first revision</i>)
2353-1963	Phenyl mercury chloride, technical
2354-1963	Ethyl mercury chloride, technical
2355-1963	Stabilized ethoxy ethyl mercury chloride concentrate
2570-1963	Methyl parathion., technical
2863-1964	Chlordane, technical
3898-1966	Zineb, technical
3900-1965	Ziram, technical (<i>first revision</i>)
~~3904~~ . 1966	Thiometon concentrates
4320-1967	Thiram, technical
4321-1967	2, 4-D technical
4344-1967	Endosulfan, technical
4345-1967	Binapacryl, technical
4451-1967	Toxaphene, technical
4929-1968	Dichlorvos , technical
4958-1968	Phosphamidon, technical
5278-1969	Dicofol, technical
5280-1969	Fenitrothion, technical
5526-1969	Coumafuryl, technical
5551-1970	Warfarin-sodium, technical
5552-1970	Warfarin , technical
6432-1972	Heptachlor, technical
7158-1973	Isobornyl thiocyanoacetate (thanite), technical
7539-1975	Carbaryl , technical
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7950-1976	Fenthion, technical
7976-1976	Phorate, technical
7977-1 976	Disulfoton, technical
7985-1976	Quintozene , technical
8024-1976	Formothion technical solution
8025-1976	Monocrotophos, technical
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